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IN MAGNETS OF 19 T AND GREATER

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## THE CHARACTERIZATION OF Nb<sub>3</sub>Sn SUPERCONDUCTORS FOR USE IN MAGNETS OF 19 T AND GREATER

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### ABSTRACT

Increased resolution of NMR spectrometry will require the use of very high field Nb<sub>3</sub>Sn superconducting magnets. Here we report the results of our investigation into mechanical and temperature effects on internal-Sn superconductors similar to those proposed for use in a 900 MHz, 21 T NMR magnet system. Thermal precompression was found to be about 0.225%, and the irreversible strain was about 0.8%. Fatigue degradation was not observed at cyclic intrinsic strains below 0.575%. Additions of reinforcing steel in cable conductors was found to reduce the critical current by as much as 50% compared to similar, unreinforced cables. Reduction of the testing temperature to 2.3 K did not increase the critical current in steel-reinforced cables to a level significantly above that of unreinforced samples.

### INTRODUCTION

Nuclear Magnetic Resonance (NMR) spectrometry is useful for the study of chemical reactions, particularly those of interest to medical and pharmaceutical research. The operating limits of these devices are a function of magnetic field, higher fields providing opportunities for increased resolution. Carnegie-Mellon University has proposed development of a 900 MHz, 21 T NMR facility with a warm bore of about 7.6 cm diameter. In support of this effort we evaluated several internal-tin superconductors and superconducting cables similar those proposed for use in this 21 T magnet. These wires are also proposed for use in a 19 T prototype coil to be tested in LLNL's High Field Test Facility (HFTF).

Due to the high current density required for 21 T operation, the proposed magnets will be constructed using cabled superconductors potted with epoxy

and bath-cooled at 1.8 K. The high Lorentz forces generated at these fields will put substantial mechanical loads on the superconducting wires. Since the magnet will be periodically de-energized for routine maintenance the effects of high strain cyclic loading were of interest. It is anticipated that the load cycles will be less than 100 over the useful lifetime of the NMR system.

We have also studied the effects of applied strain on critical current degradation, especially precompression effects caused by the inclusion of steel reinforcement in cables. It is well known that the strain sensitivity of the critical current is a strong function of field and as the operating field approaches  $H_{c2}$ , considerable degradation of  $J_c$  occurs at small applied strains. Operation of magnets at 21 T, close to  $H_{c2}$ , will require magnet designers to critically assess the effects of operational strain and precompression and account for possible degradation.

## EXPERIMENTAL PROCEDURE

Two types of internal-Sn Ti-alloyed  $Nb_3Sn$  test wires were supplied by IGC. Although we anticipate that actual magnets will probably use Ta-alloyed  $Nb_3Sn$ , due to its higher upper critical field, our initial studies have focused on this readily available material. The first type of wire tested was a 7 subelement design drawn to 0.42 and 0.92 mm diameter. The second wire, used for testing cables, was also a 7 subelement at 0.615 mm diameter. The specifications for both wires are shown in Table 1.

Table 1. Specifications of wires used in this study

|                               |                     |
|-------------------------------|---------------------|
| Seven subelement wire         |                     |
| Diameters                     | 0.42 and 0.92 mm    |
| Nb Diffusion barrier          | 4% by volume        |
| Stabilizer                    | 50% by volume       |
| Remaining non-copper fraction | 50% by volume       |
|                               | 27% Nb (1.25% Ti)   |
|                               | 19.2% Sn            |
|                               | 53.7% Cu matrix     |
| Cabled wires                  |                     |
| Diameter                      | 0.615 mm            |
| Nb Diffusion barrier          | 4.3% by volume      |
| Stabilizer                    | 62.5% by volume     |
| Remaining non-copper fraction | 33.2% by volume     |
|                               | 20.5% Nb (1.20% Ti) |
|                               | 17.2% Sn            |
|                               | 62.3% Cu matrix     |

Table 2. Test cable specifications

Cable configurations:

|               | Packing factor | Reinforcement                           |
|---------------|----------------|---|
| 1. 6 around 1 | 84%            | none                                    |
| 2. 6 around 1 | 90%            | none                                    |
| 3. 6 around 1 | 84%            | central steel wire<br>0.615 mm diameter |
| 4. 5 around 1 | 84%            | central steel wire<br>0.430 mm diameter |

Cable dimensions

1. 1.774 mm diameter
2. 1.714 mm diameter
3. 1.774 mm diameter
4. 1.517 mm diameter

The critical current as a function of applied strain and fatigue loading was measured using a system consisting of a screw-driven pull rod, digitally-controlled servo-motor and microcomputer. During testing, the pull rod and sample pass through a 12 T radial access superconducting magnet. The magnet is equipped with holmium pole pieces which raise the effective field at the specimen to 15 T. The details of this apparatus have been described previously.<sup>1, 2</sup>

Samples for strain and fatigue studies were given an internal-Sn heat treatment, found by testing, to give near optimum critical current at fields of about 14 T. This heat treatment consists of 4 steps: 200°C for 24 hours + 340°C for 48 hours + 660°C for 72 hours + 725°C for 8-12 hours depending on wire size.

Four round cables were supplied by IGC in several configurations two of which contained a steel reinforcing wire at the center of the cable pattern. The cable specifications are shown in Table 2.

The cables were mounted on 25 mm long, 50 mm diameter stainless steel spools with machined vee-grooves. Cable ends were sealed with a welding torch to prevent tin leakage. The samples were given a three step heat treatment, 200°C for 48 hours + 325°C for 24 hours + 700°C for 90 hours, in flowing argon followed by furnace cooling. This heat treatment was selected for inter-laboratory comparison of results and is not considered to be optimal.

The samples were tested on the reaction spools in a 14 T superconducting magnet oriented so that the applied field was normal to the cables. Voltage taps were placed 975 mm apart. Limited testing at temperatures below 4.2 K was achieved by pumping on a vacuum tight anticryostat which surrounded the test fixture. Temperature was monitored by carbon glass resistors placed in the helium bath near the specimens. The critical current for all samples was determined using a resistance criteria of  $1 \times 10^{-14} \Omega \cdot m$ . The critical currents reported are for the non-copper wire fraction

## RESULTS

$J_c$  as a function of strain is shown in Figure 1. The initial critical current of the 0.42 and 0.92 mm wires were 529 and 486 A mm<sup>-2</sup> respectively. For reference, a  $J_c$  versus strain curve calculated using Ekin's expression<sup>3</sup> is shown. The prestrain resulting from thermal contraction was approximately 0.225% for both wires. Critical currents ( $J_{cm}$ ) at zero intrinsic strain ( $\epsilon_m$ ) were 610 and 570 A mm<sup>-2</sup> for 0.42 and 0.92 mm diameter wires respectively. No irreversible strain degradation ( $\epsilon_{irrev}$ ) was observed below 0.8% intrinsic strain.

The results of stress-controlled fatigue tests are shown in Table 3. The results are reported as the ratio  $J_c(\epsilon)/J_{cm}$ , where  $J_c(\epsilon)$  is the critical current at the applied field and strain and  $J_{cm}$  is the critical current at field and zero intrinsic strain. Constant  $J_c(\epsilon)/J_{cm}$  indicates no cyclic degradation. Decreasing  $J_c(\epsilon)/J_{cm}$  indicates that degradation has occurred during cyclic loading. No fatigue-dependent degradation was observed until the applied strain reached 0.8% (0.575% intrinsic).

The results of 4.2 K critical current measurements of cabled conductors are shown in Figure 2. The data for each type of cable is the average of two different samples. Good agreement was found between similar samples. Cable packing factor has no apparent effect on critical current. The cables containing steel reinforcement have significantly lower critical currents than the unreinforced cables.

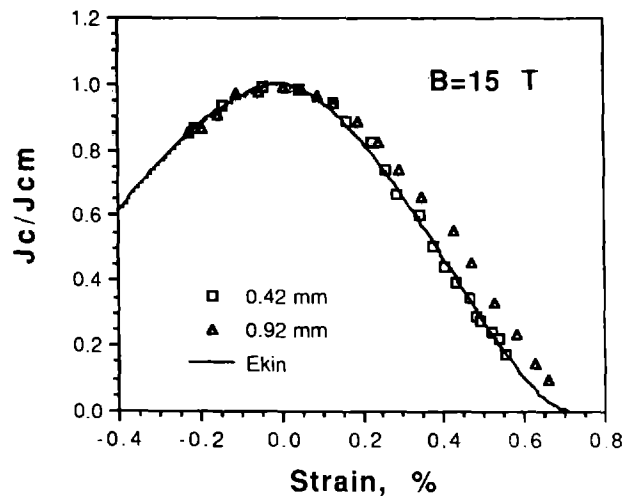


Figure 1. Normalized critical current as a function of intrinsic strain for Ti-alloyed Nb<sub>3</sub>Sn (IGC).  $J_{cm} = 610$  and  $570$  A mm<sup>-2</sup> respectively for 0.42 and 0.92 mm diameter wires. At 14 T, the reinforced 6 x 1 cables have critical currents that are approximately 50% lower than the unreinforced 6 x 1 samples.

Table 3. Fatigue effect on the critical current of internal-Sn Nb<sub>3</sub>Sn(Ti) at 15T.

| Stress<br>MPa | Strain<br>% | Cycles | $J_c(\epsilon)/J_{cn}$ |
|---------------|-------------|--------|------------------------|
| 188           | 0.4         | 1      | 0.90                   |
|               |             | 5      | 0.91                   |
|               |             | 25     | 0.91                   |
|               |             | 125    | 0.92                   |
|               |             | 1050   | 0.93                   |
|               |             | 1051   | 0.92                   |
| 210           | 0.5         | 1      | 0.86                   |
|               |             | 2      | 0.84                   |
|               |             | 50     | 0.85                   |
|               |             | 51     | 0.86                   |
| 228           | 0.6         | 1      | 0.78                   |
|               |             | 2      | 0.77                   |
|               |             | 50     | 0.77                   |
|               |             | 100    | 0.75                   |
|               |             | 101    | 0.76                   |
| 245           | 0.7         | 1      | 0.71                   |
|               |             | 2      | 0.72                   |
|               |             | 50     | 0.71                   |
|               |             | 51     | 0.70                   |
| 283           | 0.8         | 1      | 0.56                   |
|               |             | 2      | 0.53                   |
|               |             | 7      | 0.46                   |

Limited studies of temperature effects have been completed. The results of testing reinforced 6 × 1 specimens are shown in Figure 3. Each data point is the average of tests on two separate specimens. Comparison of Figures 2 and 3 shows that reducing the operating temperature of the reinforced samples to 2.3 K yields critical currents that are comparable to the unreinforced samples at 4.2 K.

## DISCUSSION

A thermal precompression of 0.225% is similar to that seen in other wires and is encouraging. The intrinsic strain to  $\epsilon_{irrev}$  is comfortably high for magnet designs that emphasize high fields and accompanying high stresses. As anticipated, there was reasonable agreement with strain-induced  $J_c$  degradation

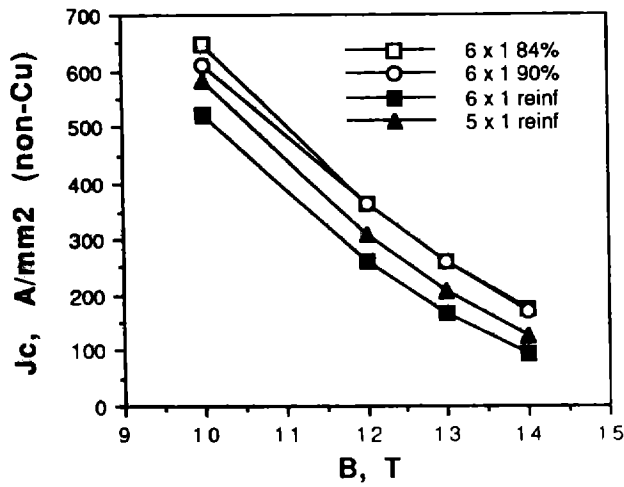


Figure 2. Critical current vs. field for cabled superconductors at 4.2K.

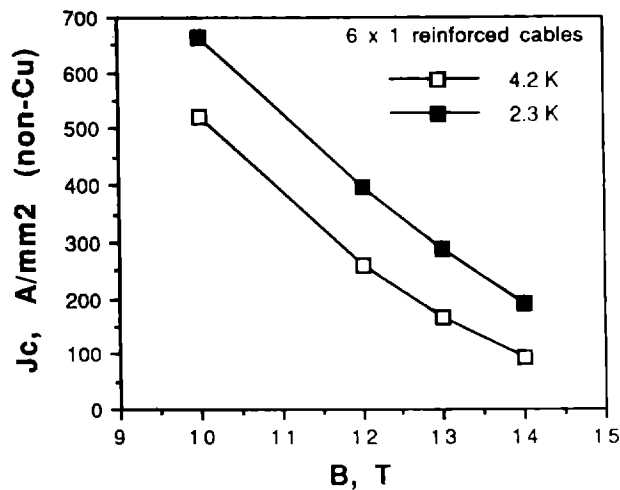


Figure 3. Effect of temperature on the critical current of reinforced 6 x 1 cables.

predicted using Ekin's formalism. The slight difference seen between the two wire diameters may result from inaccuracy in measuring applied strains during testing.

Optimal magnet design would encourage the acceptance of high operating loads in order to minimize the magnet space needed for structural elements such as the magnet case or reinforcing epoxy insulation. For steel structures, prudent design would limit strains to about 0.3%, which is equivalent to about 1/2 the yield strength of high-strength cryogenic steels such as 316 LN or JBK-75. Additionally, strains above 0.25% may be considered impractical for organic insulators, such as G-10 or other epoxies. Designs which



approach these strain limits have the advantage of relieving a large fraction of the thermal precompression. For the wires studied here, for which the precompression is about 0.225%, these strain limits would leave the conductor near zero intrinsic strain. For operation at extremely high fields where strain degradation is excessive, this represents an ideal magnet design scenario.

The performance of these wires under conditions of fatigue will not impact design of the 19 T prototype or 21 T NMR magnets where the number of load cycles is small. For the limited number of cycles tested no degradation was observed until applied strains of 0.8% were reached. This corresponds to 0.575% intrinsic strain which provides a comfortable margin between  $\epsilon_{irrev}$  and the optimal operating strain  $\epsilon_m$ .

Comparison of the critical currents of reinforced and unreinforced cables points out one possible pitfall in this approach to stress management. The unreinforced 6 x 1 cables clearly have the highest critical currents while the 5 x 1 and 6 x 1 reinforced cables follow in order. It is interesting to note that the 5 x 1 and 6 x 1 reinforced cables are 9 and 14% steel by volume. Hence, the degradation seems to scale with volume fraction of steel in the conductor. At this point the evidence is not irrefutable, however, the indication is clear that incorporation of steel reinforcement may result in increased thermal precompression hence lower  $J_c$ 's. The magnitude of this result is surprising since the cables were soldered to stainless steel drums which, by themselves, should introduce a high precompression on cooling. This indicates that co-winding steel and superconductor leads to very effective coupling and transmission of strain. If such precompression were present in an actual magnet only a small portion could be relieved by operating strains. Higher operating strains, giving near-zero intrinsic conductor strain, would not be possible since structural material operating limits would be exceeded. Quantitative estimates of the degree of precompression in the cables is complicated by soldering to the steel test fixtures.

In actual magnets, the cable space is filled with epoxy which has a low 4 K modulus ( $\approx 5$  GPa). To what extent reinforcing core precompression occurs in that case, then, is uncertain. Further testing under appropriate conditions is advisable. In addition, testing of unsoldered, unfilled cables would be interesting to determine the magnitude of direct mechanical interaction that exists between the reinforcement and superconductor. This may have consequence to cable-in-conduit conductor (CICC) design. A previous study of the effects of different core elements<sup>4</sup> proved inconclusive.

Data at temperatures below 4 K are still being collected at this time, and the limited information presented in Figure 3 is still inconclusive. However, it is interesting to note that a 1.9 K temperature reduction in the reinforced samples yields critical currents that are only slightly higher than the unreinforced samples at 4.2 K. Clearly, unreinforced cables have a significant advantage.

## CONCLUSIONS

Thermal precompression in the 7 subelement Ti-alloyed internal-Sn wires tested was 0.225%. Irreversible strain degradation was not observed until the applied strains exceeded 0.8% intrinsic strain. Cyclic fatigue at applied intrinsic strains of less than 0.8% did not cause degradation of critical current while cyclic intrinsic strains greater than .575% resulted in immediate fatigue degradation of  $J_c$ .

Small changes in the conductor packing fraction showed no measurable effect on the  $J_c$  of small cables. Inclusion of steel reinforcement, on the other hand, was found to reduce  $J_c$  by as much as 50% at 14 T, presumably due to thermal precompression. This effect scaled with the volume fraction of steel included in the cable.

Decreasing the testing temperature was found to affect large increases in  $J_c$ , however cables containing 14% steel reinforcement (6 x 1) only slightly exceeded unreinforced cable critical currents even at 2.3 K. Steel reinforcement may potentially be unacceptable for high field magnets due to precompression effects.

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